

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Application of :	§	Before the Examiner
Zhao et al.	§	James E. McDonough
Serial No. 10/692,827	§	Group Art Unit No. 1755
Filed: October 24, 2003	§	Attorney Docket Number: 2002B130A/2
For: Late Transition Metal	§	Confirmation Number 9211
Catalysts for Olefin	§	
Oligomerizations	§	
Customer No.: 23455	§	Date: July 7, 2008

Commissioner for Patents  
P. O. Box 1450  
Alexandria, VA 22313-1450

**Declaration Under 37 C. F. R. § 1.132**

Dear Sir:

This affidavit is submitted to offer a showing that:

- 1) Claims 1-20, 27-31 and 36-39 are both novel and non-obvious over Sumi (US 6,323,353) in view of Baardman (US 5,658,982) in further view of Qian (*Synthesis and Polymerization Behavior of Various Substituted Half-Sandwich Titanium Complexes Cp'TiCl<sub>2</sub>(OR\*) as Catalysts for Syndiotactic Polystyrene*, J. Mol. Cat. 208, 2004, 45-54.);
- 2) Claims 1-20, 27-31 and 36-39 are both novel and non-obvious over Buchwald (US 6,307,087) in view Baardman in further view of Qian;
- 3) Claims 1-20, 27-31 and 36-39 are both novel and non-obvious over Zhang in view of Baardman in further view of Qian;
- 4) Claim 40 is both novel and non-obvious over any of Sumi, Buchwald, or Zhang in view of Baardman in further view of Piekarski (US 3,991,259).

This affidavit is submitted concurrently with a response after final rejection to the final office action dated March 4, 2008, provided as the submission required under 37 CFR § 1.114 for a Request for Continued Examination.

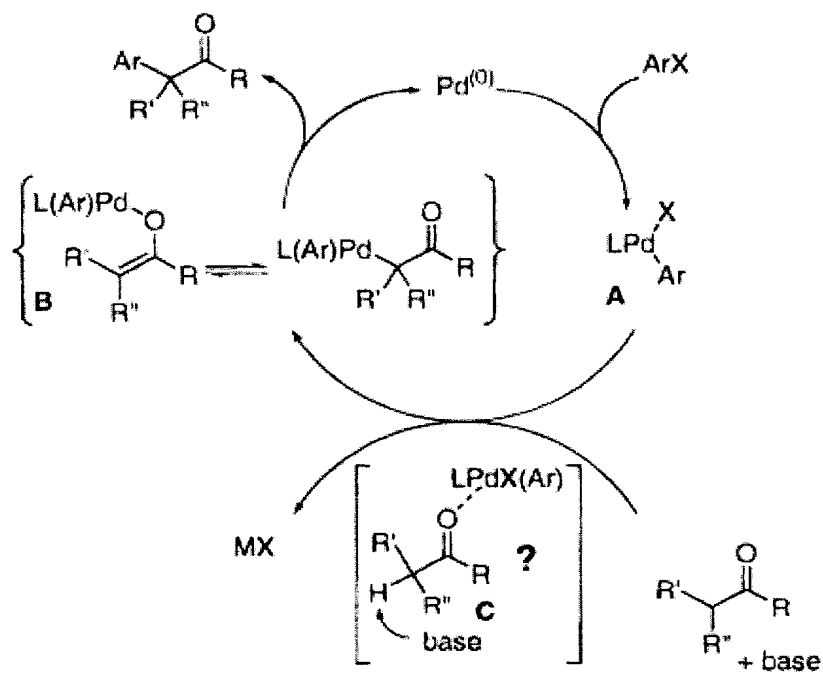
I, Jo Ann Marie Canich, Ph.D. declare that:

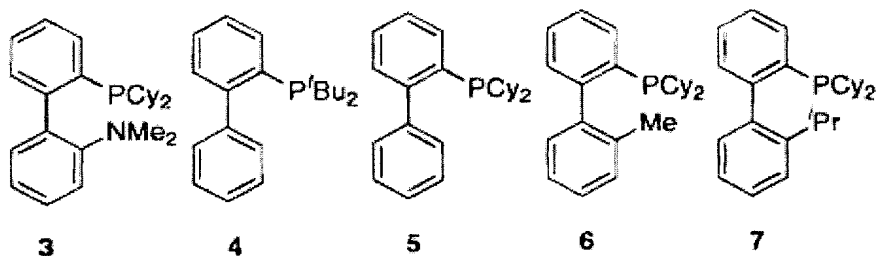
1. I am a citizen of the United States residing in Houston, Texas.
2. I am a co-inventor of the above-referenced patent application referred to herein as the '827 Patent Application.
3. I have been continuously employed by ExxonMobil Chemical Company, (and its predecessor Exxon Chemical Company) since 1987 as a research scientist working in the area of olefin catalysis. I have over twenty years of direct experience in the olefin catalysis art and, among other things, have recently published a book (co-editor) in the area of olefin catalysis (*Stereoselective Polymerization with Single-Site Catalysts*, Eds. Baugh & Canich, CRC Press, Boca Raton, FL, 2008). I have won multiple awards both inside my company and from industry for my work in the olefin catalysis field. Additional information regarding my technical and professional background is attached as a *Curriculum Vitae*.
4. I have read the office action dated March 04, 2008 and have read each of the references cited therein, e.g. Sumi, Buchwald, Zhang, Qian, Baardman and Piekarski.
5. In my opinion one of ordinary skill in the olefin catalysis art reading Buchwald, Sumi and Zhang would find that Sumi, Buchwald and Zhang are directed to catalysts useful to make small molecules via metal mediated coupling reactions, but not polymerization or oligomerization catalyst compounds.

It is further my opinion that Buchwald does not disclose the pre-catalyst compounds or polymerization catalysts used in my patent application, and only discloses the use of the ligand in combination with a metal and a base such as an alkoxide, amide, phosphate, or carbonate. While the Examiner suggests that the reaction of the ligand with a metal will produce the pre-catalysts of my invention, this is not indicated in or supported by Buchwald. In fact, if one refers to *Journal of the American Chemical Society* 2000, 122, 1360 (Fox et al.), Fox et al. shows the proposed reaction scheme (Scheme 1, page 1366 - also shown below) for a small

molecule coupling reaction where the ligand, L, is shown in the chart on page 1362. The ligand labeled **3** can perform as an ancillary ligand where Y is biphenyl (referring to the formula in Claim 2 of the '827 application). On page 1366, Fox et al. states that in comparison to previous studies, that they believe the Pd:phosphine ratio when using ligands **4-7** for the reaction intermediates (**A** and **B**) is 1:1 (i.e. only one electron donating atom, in this case phosphorous, is needed). Note that ligands **4-7** are mono-dentate phosphorus ligands. Additionally, similar evidence suggests that the binding of the dimethylamino group of ligand **3** to Pd is not essential for the catalytic process and, in fact, may not occur at all. This is indicated by the similarities in the catalysis involved when using **3**, **6** and **7** in the Pd-catalyzed ketone arylations reported in the paper, in addition to Suzuki and amination reactions reported in references within. Note that ligands **6** and **7** do not contain the dimethylamino group as compared to ligand **3**. Hence, it would be speculative at best to assume that the reaction between ligand **3** and a metal would form the polymerization pre-catalyst of my invention, when the chemistry in small molecule coupling reactions suggests otherwise.

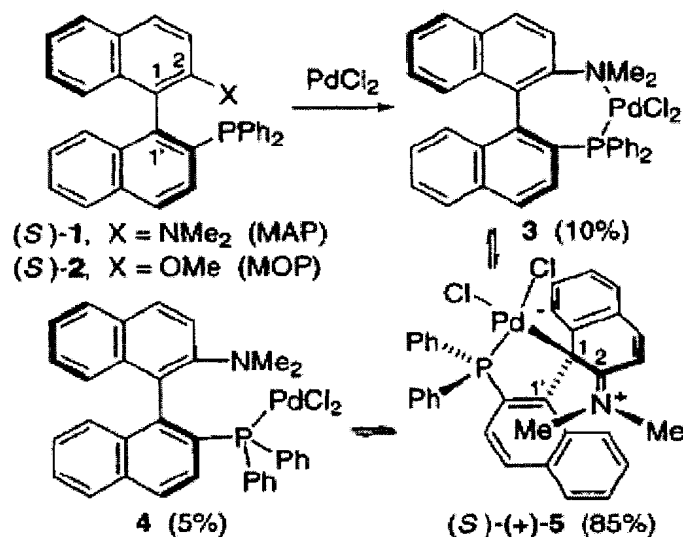
**Scheme 1**





Fox et al. also states that it is possible that the interaction between the metal and the ligand in the Pd(II) intermediates complexes (**A** and **B**) is similar to those reported for the MOP and MAP ligands reported in *Journal of the American Chemical Society* 1999, 121, 7714 (Kocovsky et al.). Kocovsky et al. uses the binaphthyl ligand also reported in Sumi (but not an ancillary ligand in my application). Scheme 1 of Kocovsky et al. is shown below (p 7714). Kocovsky et al. indicates an equilibrium between species **3** (10%), **4** (5%) and **5** (85%) in solution, however, they were only able to isolate compound **5**, suggesting the preferred state of the molecule is complex **5**. This is based not only on the ability to isolate complex **5** in crystalline form which was structurally characterized by X-ray crystallography, but also on the solution equilibrium mixture being 85% complex **5**. Note that complex **5**, and also complex **4**, does not have a metal-nitrogen bond as required of my ancillary ligand in the claims. Again, it would be speculative to assume the reaction between the ligand of Buchwald would react to make the complexes of our application verses assuming other chemistry as suggested by Kocovsky.

**Scheme 1**



One should also note that the catalysis scheme for making small molecules via metal mediated catalysis as shown in Fox et al.'s Scheme 1 involves (1) starting with a  $\text{Pd}(0)$  complex and oxidizing it to  $\text{Pd}(\text{II})$  by  $\text{ArX}$  to form intermediate **A**; (2) forming an interaction between intermediate **A** and an electron donor (a ketone in this case) in the presence of a base to form intermediate **C**; (3) abstraction of an acidic proton by the base to form intermediate **B**; and finally (4) reduction of intermediate **C** ( $\text{Pd}(\text{II})$  down to  $\text{Pd}(0)$ ) and formation of an arylated ketone. This is a catalytic cycle that involves multiple Pd species, and requires the ability to oxidize the metal to a higher oxidation state in order for the chemistry to occur, and then reduce the Pd back to  $\text{Pd}(0)$  to regenerate the catalyst and to "release" the new compound. The coupling mechanism for Buchwald is analogous to that represented by Fox et al. This is why Buchwald states in Column 32, line 7-8 that "the metal center is desirably in the zero-valent state or is capable of being reduced to metal (0)". The nickel and palladium species in my application are  $\text{Ni}(\text{II})$  and  $\text{Pd}(\text{II})$  and the catalytic cycle for olefin polymerization/oligomerization does not involve changing the oxidation state of the catalyst precursor, or of the catalyst species during the polymerization / oligomerization reaction.

For similar reasons, Sumi and Zhang do not disclose the pre-catalyst compounds or polymerization catalysts used in my patent application. Like Buchwald, both are directed toward small molecule synthesis using metal mediated catalysis.

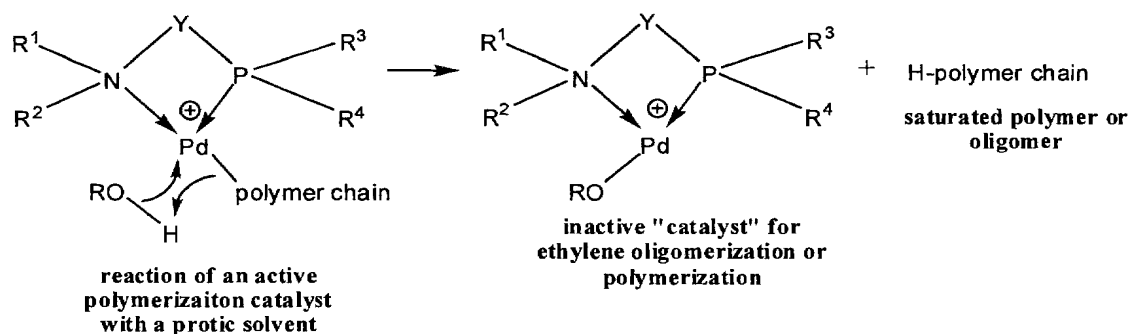
6. It is my further opinion that one of ordinary skill in the art would not combine a *polymerization catalyst activator* with the various compounds of Sumi, Buchwald and Zhang, because they do not need a *polymerization catalyst activator* to achieve their purpose of making small molecules using metal mediated coupling chemistry.

7. It is my further opinion that one of ordinary skill in the art reading Baardman would find Baardman is directed to certain Group 10 metal compounds used in combination with a borane activator to polymerize carbon monoxide and olefins in a variety of polymerization environments. Baardman shows that by increasing the amount of tris(perfluorophenyl)boron during the propylene/CO polymerization that the activity of the copolymerization can be enhanced. In this, Baardman teaches that you can “reactivate” your catalyst with additional amounts of tris(perfluorophenyl)boron. It is well known in the literature that oxidants are commonly added to this type of catalyst system to re-oxidize the Pd from the inactive Pd(0) complex to the active Pd(II) complex. It is also known that the presence of the oxidant can be avoided by using fluorinated alcohols as the polymerization solvent, hence, one of ordinary skill in the art would expect that a perfluorinated borate would act in a similar manner. See *Organometallics* 2000, 19, 3435.

While Baardman also teaches that a variety of diluents can be used, Baardman indicates that the liquid diluent to be used is one in which the copolymer to be prepared forms a suspension (i.e. is insoluble or virtually insoluble). Particularly preferred are mixtures of aprotic diluents and protic diluents because these provide a further improvement in maintaining the polymerization rate at the initial level. Protic diluents include lower alcohols such as methanol and ethanol while aprotic diluents can include ketones, chlorinated hydrocarbons and aromatics. (see col. 5, line 55 to col. 6 line 3).

8. It is my further opinion that one of ordinary skill in the art would not combine Baardman and Sumi, Buchwald or Zhang because polymerization catalyst activators are not needed to make the small molecules in Sumi, Buchwald and Zhang. In addition, some of Baardman's environments, such as the use of protic diluents would negatively affect the catalysts in Sumi, Buchwald and Zhang assuming such catalysts were being used for olefin oligomerization or polymerization (there are no teachings for such chemistry, as Sumi, Buchwald and Zhang are all directed toward

small molecule synthesis involving metal mediated coupling chemistry). For olefin polymerization or oligomerization, a protic diluent would be expected to act as a chain termination agent forming a saturated oligomer or a polymer with a saturated end-group. The catalyst would be deactivated in the process. An activator such as tris(perfluorophenyl)boron would not be capable of reactivating the catalyst for olefin oligomerization or polymerization. This reaction is illustrated below:



This is not an issue with the catalyst system of Baardman, as CO (but not ethylene or propylene or any alpha-olefin) would be capable of inserting in the Pd-OR bond. Thus, in my opinion, the teaching from Baardman, assuming one would think of using Buchwald, Zhang or Sumi for polymerization catalysts, would in fact lead to an inactive system for the polymerization or oligomerization of olefins. For a fundamental understanding of olefin/CO copolymerizations, see Nozaki, K, *Tacticity in Ethylene/Carbon Monoxide/Vinyl Co- and Terpolymerizations*. In *Stereoselective Polymerization with Single-Site Catalysts*; Baugh, L.S., Canich, J.M., Eds.; CRC Press: Boca Raton, FL, 2008; pp 577-591.

9. Further, it is my opinion that if one of ordinary skill in the art were to combine Baardman's activators with Sumi, Buchwald and Zhang's catalysts, one would expect the large excess of borate to be deleterious as it would inhibit the catalytic cycle described above in paragraph 5 (Fox et al. Scheme 1). Thus one skilled in the art would not add an acidic borate to a reaction scheme that is facilitated by a base. Even if enough of a strong base were added to neutralize the acidic borate, at most, small molecules would be produced because it is generally the catalyst that controls the product made and one would expect products similar to those produced in Sumi, Buchwald and Zhang.

10. It is my further opinion that halogen ligands and hydrocarbyl ligands are not equivalent in all catalyst systems at all times. First halogen ligands are considered electron withdrawing, whereas hydrocarbyl ligands are typically

considered electron donating. Second, in many systems, a borate activator in combination with a halogenated catalyst precursor (e.g.  $\text{Cp}_2\text{ZrCl}_2$ ) produces an inactive system while an alkylated precursor (e.g.  $\text{Cp}_2\text{ZrMe}_2$ ) will likely, but not always, produce an active system. Thus, halogens and hydrocarbyls are not equivalent, particularly in borate activator containing systems.

Additionally, Qian is directed to polymerizing styrene, not to oligomerizing or polymerizing alpha-olefins as in my application. As such, a trend seen in Qian can not necessarily be equated with a different type of polymerization process. Qian is focused on changing the  $\text{Cp}'$  or  $\text{R}^*$  of  $\text{Cp}'\text{TiCl}_2(\text{OR}^*)$ . Qian states, "In order to study how the changes of pi-donor ligand and sigma-donor ligand affect the catalytic activity and the properties of s-PS, this work aims to design and prepare a series of  $\text{Cp}'\text{TiCl}_2(\text{OR}^*)$  complexes with different substituents in Cp and  $\text{OR}^*$ , and then to apply them to the production of s-PS." Since  $\text{Cp}'\text{TiCl}_3/\text{MAO}$  is known to be a styrene polymerization catalyst, perhaps the Examiner is comparing the  $\text{OR}^*$  of Qian to Cl, but  $\text{OR}^*$  is not a hydrocarbyl, nor is it an abstractable ligand/leaving group in this catalyst system. The conclusion in Qian states, "through the polymerization testing we can conclude that the environment of cyclopentadiene plays a major role in the series of  $\text{Cp}'\text{TiCl}_2(\text{OR}^*)/\text{MAO}$  catalytic systems, and that the influence of  $\text{OR}^*$  is relatively weak." Qian's conclusion does not mean that  $\text{R}^*$  has little effect, it means that in comparison to changing the substituents on the Cp ring, it has little effect for the polymerization of styrene.

Furthermore, it is commonly known in polymerization catalysis that changing the leaving group can affect catalyst activity, polymer molecular weight, and when applicable, polymer tacticity. For example, Kaminsky et al. reported that the styrene polymerization activity of fluorinated complexes  $\text{Cp}'\text{TiF}_3/\text{MAO}$  ( $\text{Cp}' = \text{Cp}, \text{Cp}^*, \text{MeCp}, \text{EtMe}_4\text{Cp}$ , etc.) was about 30-40 times higher than with analogous  $\text{Cp}'\text{TiCl}_3/\text{MAO}$  systems. The polymer molecular weight, melting point and syndiotacticity were also significantly higher. This higher activity and syndioselectivity of the fluoride catalysts as compared to the chloride analogs is attributed to a greater number of more stable Ti(III) active sites where the fluoride would be expected to better stabilize the Ti(III) oxidation state. See *Macromolecules* 1997, 30, 7647-7650. Similarly, in ethylene polymerization, McCullough et al. in US 6,632,901 report higher activity with  $\text{Cp}'_2\text{ZrF}_2$  is used as compared to  $\text{Cp}'_2\text{ZrCl}_2$



(Cp'=1,3-Me,BuCp, Me<sub>4</sub>Cp, n-PrCp, etc.) Similarly, Pedeutour et al. (*Journal of Molecular Catalysis A: Chemical* 2001, 176, 87-94) studied the effect of the leaving group for rac-Et(Ind)<sub>2</sub>ZrX<sub>2</sub> where X, the leaving group, was Cl, Me, NMe<sub>2</sub> or CH<sub>2</sub>Ph. In this study, it was found that when X was hydrocarbyl (Me or CH<sub>2</sub>Ph) that catalyst activity was not dependent on the MAO to pre-catalyst molar ratio (Al:Zr) over the range of about 150-2000. This was not the case for the Cl leaving group where the activity differed significantly from low Al:Zr of 150 (activity = 0) to a higher Al:Zr of 2000 (activity = 1290 kg/mol-hr). This study clearly shows that for a polymerization catalyst leaving group, hydrocarbyl is not equivalent to halide. Probably more relevant is US 7,247,687 (Cherkasov et al.) which deals with late transition metal polymerization catalysts. Cherkasov et al. demonstrate that the [1,4-bis-substituted)-1,4-diaza-1,3-butadiene]nickel(II) catecholate complexes are very different catalysts as compared to the dibromide analogs. Note that the two differ in the X leaving groups - two bromides vs. a chelating catecholate ligand. The dibromide complexes are known paramagnetic and typically hydrocarbon insoluble complexes while, the catecholate analogs are diamagnetic and hydrocarbon soluble (see column 2 for a discussion on paramagnetic complexes in the Background section and diamagnetic complexes in the Summary section). Comparison of the polymerization of a catecholate based catalyst vs. corresponding dibromide based catalyst (compare entries 8-11 with C6-C7 in Table 1, column 308) shows the catecholate to give higher activity as compared to the dibromide. While this study did not compare hydrocarbyl to halide, it does show that the nature of the leaving group affects catalyst activity in late transition metal catalyzed olefin polymerizations.

Since many examples exist to show that the leaving groups in olefin polymerization catalysis can have a major affect on catalyst activity, one skilled in the art would not assume that a hydrocarbyl leaving group is the same as a halogen leaving group in a Pd based catalyst system.

11. In light of this lack of equivalence and the other statements above, it is my opinion that one of ordinary skill in the art would not combine Sumi, Baardman and Qian because firstly Qian is not directed at comparing hydrocarbyl verses halogen leaving groups, but is a study of ligand substitution on mono-cyclopentadienyl titanium catalysts for styrene polymerizations; secondly, Sumi is not directed to olefin polymerization catalysis, but rather to small molecule synthesis using late transition

metal mediated coupling chemistry; and thirdly, Baardman is directed to the use of added borate activator in late metal catalyzed olefin/CO polymerizations.

12. It is my further opinion that one of ordinary skill in the art would not combine Zhang, Baardman and Qian because of the same reasons stated in paragraph 11 since Zhang, like Sumi, is directed to small molecule synthesis using late transition metal mediated coupling chemistry.

13. It is my further opinion that one of ordinary skill in the art would not combine Buchwald and Qian because of the same reasons stated in paragraph 11 since Buchwald, like Sumi, is directed to small molecule synthesis using late transition metal mediated coupling chemistry.

14. It is my further opinion that one of ordinary skill in the art reading Piekarski, would find Piekarski is directed to early transition metal polymerization catalysts. Piekarski uses group 4-6 metals activated by a dialkylaluminum hydride-hydrogenpolysiloxane reaction product to make polyethylene. More specifically, the group 4-6 metals are  $TiX_{4-n}(OR'')_n$  or  $VX_{4-n}(OR'')_n$  or  $VOX_{3-n}(OR'')_n$ . The reaction of the dialkylaluminum hydride with the hydrogenpolysiloxane is not the support, and in fact, the patent states that the reaction product is a slightly viscous material (col. 5, lines 20-23). Supports are merely an addition to what is described in the patent. Piekarski teaches very little regarding supports, but his brief statement regarding increased catalyst activity when supported refers to the ability to lower the amount of catalyst needed so that it does not need to be removed from the polymer (col. 7, lines 18-39). This is a moot teaching for an oligomerization catalyst, because in any case, the support would need to be filtered from the liquid olefin. Additionally Piekarski gives no indication or hint that using a late transition metal catalyst with an activator on a support would produce oligomers.

15. It is my further opinion that it is not automatic that catalyst activity is increased by putting a catalyst compound on a support. There are many systems where putting the catalyst on a support actually reduces "activity". For example, if the support contains functional groups that interact with the catalyst, portions of the catalyst can actually be deactivated causing a reduction in polymerization activity. Additionally, for catalysts that have datively bonded ancillary ligands, if the support is not chosen wisely, the ancillary ligand may be displaced by stronger donor groups on

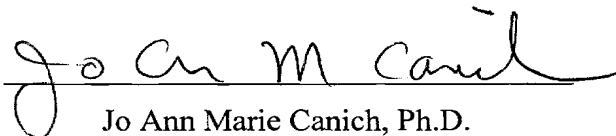
the support. These are just two of many examples where catalyst activity could be reduced by use of a support.

16. At the time the '827 application was filed, very few catalyst systems were known that combined good catalyst activity with the production of linear olefins, with a particular preference for lower alpha-olefins. Additionally, the hydrocarbon solubility of my catalyst precursor allowed for readily supporting the catalyst which can have advantages in certain processes, for example, a fixed bed process. And it is possible (but yet unproven) that the reason behind the unusual catalysis of my catalyst system, is that most four-coordinate Ni(II) complexes prefer the planar geometry since the planar ligand set allows for the d-electrons to avoid occupying antibonding orbitals. My Ni(II) four-coordinate complexes were determined to be tetrahedral in geometry meaning that d-electrons occupying the antibonding orbitals is unavoidable, hence the fact that these complexes are also paramagnetic.

17. In light of the above, it is my opinion as one of ordinary skill in the art that the foregoing clearly indicates that the catalyst described and claimed in my '827 Patent Application represents a technological break-through as perceived by those skilled in the field of transition metal catalysis.

18. *I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the patent.*

Date: July 7, 2008

  
Jo Ann Marie Canich, Ph.D.

# CURRICULUM VITAE

## JO ANN MARIE CANICH

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### **Industrial Positions:**

- ExxonMobil Chemical Company – Core Technology
  - Product Technology, Organometallic Catalysis Group (11/07-present)
    - Team Leader - Block Copolymers Team 1/08-present
  - Catalysis Process R&D, Organometallic Catalysis Group (1/07-11/07)
- Univation Technology: Catalyst R&D (1/04-1/07)
- Exxon or ExxonMobil Chemical Company – Core Technology
  - Catalysis Process R&D, Organometallic Catalysis Group (6/02-12/03)
    - Intellectual Property Coordination (12/02-12/03)
  - Polymer Science Division, Catalysis & Process Science Group (5/99-5/02)
  - Polymer Science Division, Solid State and Molecular Modeling Group (1/96-4/99)
    - Team Leader - Exploratory Catalyst Team 1/96-12/97
    - Team Leader - Solution Metallocene Elastomers Team (1/98-12/98)
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### **Awards:**

- ExxonMobil Chemical Company - CPR&D Research Award 2003
- American Society of Patent Holders Distinguished Corporate Inventors 1998
- Exxon Chemical Polymers Technology Award 1997
- Exxon Chemical, Baytown Polymers Center, Extra Mile Award 1995
- Exxon Chemical, Polymer Science, Where the Tire Meets the Road Award 1990
- Iota Sigma Pi National Anna Louise Hoffman Award 1987

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Ph.D. Inorganic Chemistry (8/87)

Advisor: F. A. Cotton

Thesis: Oxidative Addition Chemistry of Group V and VI Metal Dimers

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M. S. Chemistry (6/84)

B. S. Chemistry (6/81)

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Thesis: Derivatives of Trifluorosulfur Pentafluoride and F-(tert-butyl)  
Hypochlorite

### **Publications:**

*Stereoselective Polymerization with Single-Site Catalysts*; Baugh, L.S., Canich, J.M., Eds.; CRC Press: Boca Raton, FL, 2008.

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